## PATENT APPLICATION OF

# Jennifer Hay

### ENTITLED

DETECTING A SIGNIFICANT EVENT IN

EXPERIMENTAL DATA AND USE OF SUCH

FOR DETECTION OF ENGAGEMENT DURING

A MECHANICAL TEST

# DETECTING A SIGNIFICANT EVENT IN EXPERIMENTAL DATA AND USE OF SUCH FOR DETECTION OF ENGAGEMENT DURING A

### 5 **MECHANICAL TEST**

10

The present application is based on and claims the benefit of U.S. provisional patent application Serial No. 60/444,446, filed February 3, 2003, the content of which is hereby incorporated by reference in its entirety.

### BACKGROUND OF THE INVENTION

Sensitive, accurate, and consistent detection of significant events in experimental data is important capability for a myriad of applications. 15 For example, in experiments or tests that measure mechanical properties of materials, it is often important to measure a significant event known as the engagement point. For contact-type tests typically to measure a mechanical property such as hardness, and which typically utilize an indenter, 20 the engagement point is the point at which the indenter first contacts and begins to apply force to the test surface. For tensile tests measuring, for example, stress and strain, the engagement point is 25 the point at which all slack has been removed from the test apparatus and test sample, and any further movement in the same direction begins to stress and strain the test sample. In another example, it can be desirable to detect changes in temperature from a

baseline in a particular process or environment. Often it is difficult to detect a significant event using a simple threshold, especially when measurement changes are gradual. A system and method of accurately, sensitively, and consistently detecting significant events in experimental data would have significant utility.

### SUMMARY OF THE INVENTION

One aspect of the present invention includes a computer readable medium that includes computer executable instructions for detecting a significant event in measurement data. Measurements are processed to calculate values indicative of multiples of a standard deviation. The point of onset of the significant event is calculated as a function of the calculated values.

Another aspect of the present inventions also includes detecting the significant event by receiving a data series indicative of test measurements as a function of a variable. At least one processed series is generated from the data series. A first point and a second point are identified on at least one of the processed series. The significant event is then calculated based on at least one of the two points.

Other aspects of the present invention also includes associated methods and testing systems adapted to determine a significant event using methods of the present invention described herein.

20

25

### BRIEF DESCRIPTION OF THE DRAWINGS

- FIG. 1 is a schematic diagram of an environment where the present inventions can be practiced.
- FIG. 2 is a schematic diagram of a computing environment useful in practicing the present inventions.
  - FIG. 3 illustrates a block diagram of an embodiment of the present inventions and illustrates broad concepts helpful in understanding the present inventions.
  - FIG. 4 illustrates a block diagram of an aspect of the present invention.
  - FIG. 5 is a flowchart illustrating a method in accordance with an aspect of the present invention.
- FIG. 6 is a graph of measured force as a function of displacement.
  - FIG. 7 is a graph of a second derivative of the graph illustrated in FIG. 6.
- FIG. 8 is a graph of processed signals as a 20 function of time generated in accordance with the present inventions.
  - FIG. 9 is an enlargement of FIG. 6 and illustrates a point corresponding to a significant event calculated in accordance with the present inventions.

25

30

10

### DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

Referring now to FIG. 1, there is shown a schematic diagram of a testing system, such as an indenter testing system used to measure mechanical properties of materials. Testing system 100 is

modified in accordance with the present inventions for testing a sample of material 105.

5

10

15

20

25

Sample 105 is placed at a known location on computer controlled X-Y table 107 with the surface to tested facing up. Electromagnetically arrangement 109 is provided, indenter which positioned over the sample 105. Indenter 109 includes current driven load coil 111 activated by application of electrical current from computer controlled variable current source 117 to move the probe tip 115 downward into contact with sample 105. Once tip 115 contacts sample 105, a pre-selected force pattern is applied to indenter 109 by the programmed variation of the current applied to drive coil 111. In some embodiments, probe tip 115 can be in the form of a typical triangular pyramidal diamond probe with an end radius of about 500 Angstroms.

Current source 117 is controlled by system 119, which also controls X-Y table 107. computer Displacement of probe 113 is measured by capacitive displacement gage 121, whose output is connected to DC displacement detector 123. Detector 123 digitizes the DC displacement signal, which is fed through a first digital voltmeter 125 to an input of computer 119 for further processing in accordance with the present inventions. Voltmeter 125 can provide a calibrated readout of probe displacement to an operator during testing procedures, if desired.

Force applied to sample 105 through indenter probe 30 115 is monitored by DC current detector 127, which

senses the DC drive current applied to load coil 111. The DC load current is digitized by detector 127 and fed through a second digital voltmeter 129 to a further input of computer 119 for further processing according to the present inventions. In some embodiments, computer 119 can be connected to a mass storage device 131 where data and system operating parameters are stored.

5

10

15

20

25

. 30

Using testing system 100 as described above, a sample 105 is positioned at a known location on X-Y table 107 and programmed computer 119 is signaled to start a timed test and data gathering procedure. Generally, computer 119 is programmed to perform one or more prescribed indentation tests by automatically causing single or multiple indentations at designated locations on sample 105. Probe 115 is lowered at a very slow rate until contact is made with sample 105.

During the test procedure, computer 119 records measurement data, such as force taken from DC current (load) detector 127 and probe displacement taken from DC displacement detector 123. Measurement data can be used later to determine mechanical properties of sample 105. Additionally, measurement data such as values associated with measured force, displacement, and time can be processed using the present inventions to determine significant events occurring during the test procedure, such as when probe tip 115 "engages" sample 105 during the indentation procedure.

FIG. 2 and the related discussion provide a brief, general description of a suitable computing environment

in which the present inventions can be implemented. Although not required, testing system 100 and the present inventions can be described, at least in part, in the general context of computer-executable instructions, such as program modules, being executed computer 119, 219. Generally, program modules include routine programs, objects, components, structures, etc., which perform particular tasks or steps or implement particular abstract data types of the present inventions. Program modules and associated methods are described below and illustrated using block diagrams and flowcharts. Those skilled in the art can implement the block diagrams and flowcharts computer-executable instructions. Moreover. skilled in the art will appreciate that the inventions practiced with other computer system multi-processor configurations, including systems, networked personal computers, mini computers, frame computers, and the like. The inventions may also practiced in distributed computing environments where tasks are performed by remote processing devices that are linked through a communications network. In a distributed computer environment, program modules may be located in both local and remote memory storage devices.

10

15

20

25

30

Computer 219 illustrated on FIG. 2 (similar to computer 119 illustrated on FIG. 1), comprises a conventional personal or desktop computer having central processing unit (CPU) 232, memory 234 and 236, system bus which couples various system

components, including memory 234 to CPU 232. System bus 236 may be any of several types of bus structures including a memory bus or a memory controller, a peripheral bus, and a local bus using any of a variety of bus architectures. Memory 234 includes read only memory (ROM) and random access memory (RAM). A basic input/output (BIOS) containing the basic routine that helps to transfer information between elements within computer 230, such as during start-up, is stored in ROM. Storage devices 238, such as a hard disk, a floppy disk drive, an optical disk drive, etc., are coupled to system bus 236 and are used for storage of programs and data. It should be appreciated by those skilled in the art that other types of computer readable media that accessible by a computer, such as magnetic cassettes, flash memory cards, digital video disks, random access memories, read only memories, and the like, may also be used as storage devices. Commonly, programs are loaded into memory 234 from at least one of storage devices 238 with or without accompanying data.

10

15

20

25

30

Input device 240 such as a keyboard, pointing device (mouse), or the like, allows a user to provide commands to computer 230. Monitor 242 or other type of output device is further connected to system bus 236 via a suitable interface and provides feedback to a user.

Control signals 217 output to testing system 100 comprising current source 117 and X-Y table 107 (illustrated in FIG. 1) based on program modules

executed by computer 119, 219 and through suitable interface 244. Interface 244 receives output signals 221 indicative of displacement from voltmeter 125 and signals 221 indicative of force from voltmeter 129.

- 5 Response signals 221 are processed based on program modules executed by computer 119, 219 to calculate or approximate, for example, material properties or significant events occurring during the testing procedure, such as engagement point.
- 10 FIG. 3 illustrates a block diagram of embodiment of the present invention, which can be implemented with a computer such as computer 119, 219 executing instructions stored on a computer-readable medium. Modules, sub-modules, and associated steps 15 for carrying out such instructions are described Those skilled in the art will readily understand that the illustrated modules, sub-modules, and steps can be combined or divided as desired. It is noted, that all steps of the present `inventions 20 be performed after the test procedure experiment is completed. Optionally, some steps can be performed during the actual test procedure. Also, variables x, y, z, t, etc. all represent series of values that are controlled, sensed or measured during 25 the test procedure or experiment, or series that are calculated or derived from such. Examples of such series include force y, displacement x, time t, reading number, stress, strain. flow temperature, and the like. It should be noted that x, 30 y, z, and t may actually represent any experimental

series. It should further be noted that x, y, z, and t can each be independent or dependent series. For example, in a testing system that measures mechanical properties of materials, where force is controlled and displacement is a sensed response, y can represent force and t can represent displacement. Finally, it should be noted that each time the order (n) changes, the series represented by t may also change.

5

10 In FIG. 3, broad modules or steps that helpful in understanding the present inventions are illustrated. Testing system 100 (illustrated on FIG. generates signals indicative of measurements, variables, or data 302 such as x, y, z, t described above. Measurements 302 are received as input to 15 significant event determination module 300, which comprises pre-processing module 304, determination module 306, and optionally, uncertainty module 308. Pre-processing module 304 receives and processes in accordance with the 20 measurements 302 inventions to generate values used by determination module 306 to determine or detect events that are significant, such as engagement point S of probe tip 115 and sample 105 (illustrated on FIG. 1). Notably, 25 such an engagement point has values associated with variables such as force, displacement, and time at engagement. Significant event determination module 300 measurements generates or values for the variables or parameters at engagement point S, and optionally, an uncertainty as indicated at 310. 30

FIG. 4 illustrates an embodiment of the present invention and generally illustrates greater detail than the embodiment illustrated in FIG. 3. convenience, the last digits of the reference numerals in FIGS. 3 and 4 correspond where possible (such as 302, 402) to indicate similar items or steps. FIG. 5 illustrates a flowchart of an algorithm or method 500 of the present invention. The blocks in 5 are labeled with letters "A" through "P" generally indicating steps of one or more methods. However, it should be noted that the indicated steps and their respective order are illustrative only and can be combined, recombined, divided, or reordered as desired.

10

25

30

15 Testing system 100 (illustrated on FIG. 1) generates values associated with or indicative of measurements or data 302, 402. Measurements 302, 402 are provided to or received by significant event determination module 400. FIG. 5 generally 20 illustrates a step of receiving measurements as start block 502 and block 504 where the series order, n is initialized.

Significant event determination module 400 includes non-zero trend detector 422, which adapted to or capable of detecting trends in measurements 302, 402. FIG. 5 illustrates a step of detecting a non-zero trend in received measurements at block 506. Significant event determination module 400 can also include pre-processing module 404, shown in dotted lines, that corresponds to pre-processing module 304 (illustrated in FIG. 3). Detector 422 can use any known means such as mathematical methods, electronic methods, curve fitting, etc. to detect a slope, change, or non-zero trend in measurements 302, 402, especially prior to the significant event to be detected. Measurements 302, 402 can include variables such as force recorded or measured as a function of at least one other variable such as displacement and/or time. In some embodiments, force is a controlled variable and displacement is a response variable being measured.

10

15

20

25

30

Generally, non-zero trends can be caused by the physical environment or the testing system itself. For example, a load cell may have some known offset, 5mN. example, Another example is а linear relationship between force and displacement, which can exist due to internal stiffness of testing system 100. In the examples provided, both the offset in force of 5mN and the internal stiffness cause a nonzero trend before the onset of the significant event or engagement point. In the present inventions, initial non-zero trend in a data measurement signal is processed by "zeroing" or generating at least one processed signal or series that scatters fluctuates about zero.

In contrast, if non-zero trend detector 422 does not detect a non-zero trend, i.e. measurements 302, 402 are scattered about zero as a function of another variable, such as displacement or time, then measurements 302, 402 can be provided to integration

module 314 directly. However, if non-zero trend detector 422 detects a pre-significant event non-zero trend in measurements 302, 402, then measurements 302, 402 are provided to zeroing module 424 for further processing. FIG. 5 illustrates a step of zeroing at block 508, which is indicated with dotted lines. The step of zeroing can be iterative as indicated at 510. The series order is advanced with each loop, pass, or iteration through zeroing module 508 as indicated at block 512.

10

Zeroing module 424 comprises compensator and/or differentiator 412 that are used to process or generate new series or signal output approximates or scatters about zero. Compensator 410 or differentiator 412 is user selectable based on 15 design criteria as illustrated at compensation/ differentiation selection block 514 illustrated in 5. In embodiments utilizing compensation, compensator 410, (assuming variable y is a function 20 t) constructs a new series by "compensating" values (generally, but not limited to, all values) of measurements 302, 402 using an analytic function  $\Psi(t)$ . FIG. 5 illustrates compensation at compensation block 516. The function  $\Psi(t)$  can be known from the 25 physical environment or it may be determined by methods such as curve fitting within some significant pre-event period using, for example, a low-order polynomial function or some other appropriate form.

Therefore, in the above example where a load 30 cell contains a 5mN offset or  $\Psi(t)=5mN$ , compensator

410 constructs a new or processed series such as  $y_1(t)$  where  $y_1(t) = y_0(t) - \Psi(t) = y_0 - 5mN$ . In the second example where a linear relationship exists between  $y_0(t)$  and displacement t, then a linear fit to pre-event data produces the linear function  $\Psi(t)$  where  $\Psi(t) = kt + b$ , k is slope, and b is the constant at t=0. Compensator 410 constructs or generates a new series  $y_1(t)$  where  $y_1(t) = y_0(t) - \Psi(t)$ , or  $y_1(t) = y_0(t) - kt - b$ .

In embodiments illustrative of differentiation, 10 differentiator 412 takes successive derivatives, usually up to a first or second derivative to construct at least one new or processed series scattering about zero. Thus, in the above example where a load cell has a 5mN offset, series  $y_0(t)$  is approximately equal to 5mN, and differentiator 412 15 generates a new series  $y_1(t)$  from  $y_0(t)$  by taking a first derivative of  $y_0(t)$  so that  $y_1(t)=y_0'(t)=0$ . Thus, the new series  $y_1(t)$  now scatters about or is approximately zero. In the second example where a 20 linear relationship exists between  $y_0(t)$ displacement t, then series  $y_0(t)$  can be expressed as  $y_0(t)=kt+b$ , where k is slope and b is a constant as above. On a first iteration through differentiator 412 (illustrated as differentiation block 518 on FIG. 25 5), differentiator 412 takes a first derivative of  $y_0(t)$  to generate a new or first processed series  $y_1(t)$ , where  $y_1(t)=y_0'(t)=k$ . This means that series y<sub>1</sub>(t) scatters about k after a first pass through differentiator 412. Differentiator 412 then loops and

generates a second derivative of  $y_0(t)$ , or a second processed series  $y_2(t)$  where  $y_2(t)=y_1'(t)=y_0''(t)=0$ .

Thus, in the second example two loops or passes through differentiator 412 are needed to generate or construct series y2(t) scattering or fluctuating about zero. It should be noted that differentiation can be accomplished by any known means, including but not limited to numerical methods, electronic methods, or fitting. curve In many instances, it can advantageous to use differentiation versus compensation. For example, with differentiation, it is not generally necessary to know the underlying functionality of series variable  $y_n$ , e.g. it is not necessary to know load offset, or precise linear relationship between y and t. It should also be noted that there can be a pre-event quadratic, cubic, or higher order polynomial relationship between y and t, which would seemingly call for third, fourth, and higher order derivative to generate a processed series scattering about zero. However, taking successive derivatives generally results in greater signal instability, which effectively limit the number of derivatives that can be taken.

10

15

20

In another embodiment, both differentiation and compensation can be used to zero input measurements. Thus, assuming there is a quadratic relationship between two variables such as force y and displacement t, then initially,  $y_0$  can be expressed as  $y_0=at^2+bt+c$  where a, b, and c are constants. Taking iterative 30 first and second derivatives of  $y_0$  yields

 $y_2(t)=y''(t)=c$ . Compensation can then be used where  $\Psi(t)=c$  so that new series  $y_3(t)=0$ . Thus, compensation and differentiation can be combined, especially to limit signal instability when a derivative greater than a second derivative is needed to zero input measurements.

Zeroing module 424 outputs or generates processed series yn also called zeroed or "derivative signal" where at least a portion of the derivative signal 10 scatters about zero, especially pre-event data. It is important to reiterate, however, that there situations where input measurements 302, 402. especially pre-event data, scatter about zero and are, therefore, not in need of compensation 15 differentiation. Thus, derivative signal 413 is intended to include a series, new or original, that scatters about zero with or without differentiation or compensation. Derivative signal 413 is provided to or received by optional integration module 414 20 further processing. The flowchart FIG. of illustrates that integration is optional at block 520. However, in most situations integration is needed to implement the present inventions. In embodiments where integration is selected, integration module 25 generates or constructs new series  $y_{n+1}(t)$  also called "integral signal" 415 by integrating series  $y_n$  with respect to t over a selected increment or period au. FIG. 5 illustrates the step of integration integration block 522, which advances the series order

at block 524. The processed series  $y_{n+1}(t)$  generated by integration module 414 can be computed as follows:

$$y_{n+1}(t) = \int_{t-t}^{t} y_n(t)dt.$$
 Eq. 1

Thus, the series  $y_{n+1}(t)$  generates values indicated at 416 for any value of t. It is noted that integration can significantly increase sensitivity to significant event onset, especially over some previous period  $\tau$ , because gradual changes are cumulative or accumulated.

In integration module 414 a period  $\tau$  can be selected so that  $\tau$  is several times greater than the period in which detection is desired. For example, if t represents time and detection is desired within 1 second, then  $\tau$  could be selected around 5 seconds. If  $\tau$  is set too large, the small offsets in  $y_n$  may be accumulated, and  $y_{n+1}(t)$  may not be centered about zero.

Optionally, integration module 414 can compute values 416 using the following equation:

$$y_{n+1}(t) = \frac{1}{\tau} \int_{t-\tau}^{t} y_n(t) dt,$$
 Eq. 2

20

25

5

10

15

which can be viewed as the mathematical expression of mean values of  $y_n(t)$  over the previous period,  $\tau$ . Integration can be accomplished by several known means, including but not limited to, numerical methods, electronic methods, or curve fitting.

Integral signal 415 having values 416 generated by integration module 414 are then output to or received by normalization module 418, which is

indicated at normalization block 526 on FIG. 5. It should be noted that normalization module 418 generates the same results on output signal 417 regardless of whether above Eq. 1 or Eq. 2 is used in integration module 414. Normalization module 418 comprises standard deviation computation module 420, which computes or generates standard deviation  $\sigma$  421 for series or values 416 of integral function 415 over some period (typically a significant interval) before the significant event. FIG. 5 illustrates computing standard deviation  $\sigma$  421 at block 528.

5

10

15

20

25

30

Normalization module 418 processes or generates a new series  $y_{n+1}(t)$  by dividing all values 416 in series  $y_n$  by standard deviation  $\sigma$  as indicated at 422. FIG. 5 illustrates the step of dividing  $y_n$  by the standard deviation  $\sigma$  at block 530 to advance the series order as indicated at block 532. Thus, the output normalization module 418 are values 424 that dimensionless and multiples of the standard deviation  $\sigma$  . These values 424 can then be used to detect events such as the point of engagement. Thus, significant events are calculated or determined as a function of these values 424 that are indicative of standard deviation. especially multiples of the standard deviation.

Normalization module 418 outputs values 424 that are received by determination module 306, 406. FIG. 5 illustrates the broad step of event determination at block 534 indicated as dotted lines and comprising several sub-blocks 536, 538, and 540. Engagement

determination module 306, 406 comprises user selected threshold settings  $h_1$  and  $h_2$  as indicated at 426. The threshold setting h<sub>1</sub> is selected as a lower bound to indicate that the significant event has not yet occurred. The threshold setting h2 is selected to indicate an upper bound or a point where significant event is assumed to have already occurred. It is generally desirable to set threshold setting  $h_2$  in consideration of a desired confidence level. Generally, if the pre-event distribution of  $y_n(t)$  is approximately Gaussian, then a threshold setting  $h_2$  in the range of 10 and 20 (i.e. where are between 10 and 20) values 424 yields results. As with  $h_2$ , threshold setting  $h_1$  should be set in consideration of the desired confidence level. Generally, selecting a threshold setting  $h_1$  at a value less than 1 (i.e. where values 424 are less than 1) yields good results.

10

15

Engagement determination module 406 then uses threshold settings  $h_2$  and  $h_1$  to determine points  $S_2$  and  $S_1$  respectively, as indicated at 428. The point  $S_2$  is determined using a processed series  $y_n(t)$  and threshold  $h_2$ .  $S_2$  is the first point in the series  $y_n(t)$  for which there is excellent evidence for the onset of the event. Thus,  $S_2$  is the first point, actual or interpolated, for which  $y_n(t)$  differs from zero by a threshold,  $h_2$ . Thus, if the threshold  $h_2$  is set at 15, then the point  $S_2$  occurs where the value of  $y_n(t)$  first exceeds 15. Once  $S_2$  has been determined,

it can be associated or correlated with points in other data series of the experiment.

Point  $S_1$  is then determined starting with the point  $S_2$  and stepping back through values in the series  $y_n(t)$ . Point  $S_1$  is the first encountered point, actual or interpolated, for which  $y_n(t)$  is within a given absolute-value threshold setting,  $h_1$ . Absolute values are used because values for series  $y_n(t)$  can be positive and negative in some situations. FIG. 5 illustrates steps of determining points  $S_2$  and  $S_1$  at block 536 and block 538, respectively.

10

15

20

25

30

Determination module 306, 406 calculates event such as engagement, herein denoted as point S or S' (illustrated in FIGS. 8 and 9) using  $S_1$ ,  $S_2$ , and any combination of data series. FIG. 5 illustrates step of calculating S, S' at block Calculation of S, S' may be accomplished in any number of ways. For example, it can be sufficient to set S to be the same point as  $S_2$ , or S can be calculated to be the first actual or interpolated point after  $S_1$ , for which  $y_n(t)$  differs from zero by a less stringent threshold  $h_s$  of, say, 4. It should be noted that in alternate embodiments, engagement point denoted as S' does not necessarily have to lie on a particular experimental trace z(t). For example, if z represents force and t represents displacement, it useful to calculate S′ as the unique intersection point of linear extrapolations of z(t)data before  $S_1$ and after  $S_2$  (as illustrated as intersection point S' on FIG. 9) in which case, S'

may have associated values of force and displacement that are not actual measured values. Once S (or S') has been determined, it can be corresponded with values in other data series for the experiment, such as temperature. FIG. 5 illustrates the step of determining S at block 540.

Significant event determination module 400 can include uncertainty module 308, 408 to calculate uncertainty  $\delta\!z$  . The step of determining uncertainty is illustrated as block 542 on FIG. 5. Uncertainty  $\delta\!z$  is the uncertainty in any sensed, response, controlled, or calculated series due to the uncertainty in the point of onset of the event. Calculation of  $\delta\!z$  can be accomplished any number of ways. A conservative estimate of  $\delta\! z$  might be obtained by calculating half the variation  $\boldsymbol{z}$ between and in  $S_1$  $S_2$ . uncertainty can be calculated as a function of S1 and extrapolations are used to obtain uncertainties can be based on uncertainties in the fitted coefficients of the extrapolations. Significant event determination module 400 generates engagement point S and uncertainty  $\delta z$  as indicated at 410, and illustrated at end block 544 on FIG. 5, that are then available to a user and/or for further processing.

# 25 <u>An example</u>

5

10

15

20

30

To demonstrate the present inventions, a hardness test was performed using a testing system similar to testing system 100 illustrated in FIG. 1. Although not required, such a testing system can include a computer-controlled AC signal generator fed to current source

117 and an AC displacement detector in line between capacitive displacement gage 121 and computer 119. Such a testing system has been referred to as a Nanoindenter XP, which is manufactured by MTS Systems Corporation of Eden Prairie, Minnesota and described in U.S. Patent 4,848,141, which is herein incorporated reference. Although not illustrated, a similar test procedure can be performed on a tensile testing system such as described in U.S. Patent No. 6,679,124, herein incorporated by reference in its entirety. In the context of a hardness test, the "significant event" of interest is engagement between an indenter or probe tip 115 and the surface of a test sample such as sample 105. Force  $(y_0)$ , displacement (x), and time (t) were recorded during the initial part of a hardness test on polycarbonate before, during, and after engagement or contact. Polycarbonate was chosen because it rather compliant material, which makes engagement detection more challenging.

10

15

20 FIGS. 6-9 are pictorial graphs that illustrate results of a test procedure performed in accordance with the present invention. However, it should be noted that although pictorial graphs are helpful understanding aspects of the present invention and can 25 be rendered if desired to the user, pictorial graphs are not necessary for processing data associated with a significant event. FIG. 6 is graph illustrates measurements of force (mN) as a function of a variable displacement (nm). Both force, displacement, and time measurements were recorded during the test 30

procedure and received by a computer such as computer 119, 219 (illustrated on FIGS. 1 and 2, respectively) for further processing in accordance with the present inventions. Graph 600 illustrates point S, indicated at 602, which is the calculated engagement point according to the present inventions. To the left of S as indicated at 604, there is no engagement. To the right of S as indicated at 606, the probe tip is engaged with the sample.

In order to obtain a series that is scattered about zero before contact, a plurality of new or processed series were generated by taking a first derivative and then a second derivative of force with respect to displacement. The second derivative was calculated using the following equation:

$$y_2(x) = \frac{d^2(y_0(x))}{dx^2}$$
 Eq. 3

It is noted that the notation  $y_2(x) = y_0''(x)$  used above is equivalent to the notation in Eq. 3. FIG. 7 is a graph 700 illustrating the results for new series  $y_2(x)$  plotted as a function of displacement x using Eq. 3. The differentiation was performed numerically using any point of interest and several data points backward (to the left). In this context, repeated differentiation is advantageous because it can be done in real-time with no a priori information regarding the pre-engagement functional relationship between force and displacement. (In other words, one doesn't need to know the offset or the stiffness of the

testing instrument.) The disadvantage is that repeated numerical differentiation of experimental data tends to produce series that are progressively more "scattered."

FIG. 8 is graph 800 illustrating series  $y_3$  and  $y_4$  as a function of another variable time t rather than displacement x. In order to illustrate the advantage of integration such as illustrated by integration module 414 on FIG. 4 and block 522 in FIG. 5, processing was performed with and without integration. Thus,  $y_3$  indicated at 802 is the series without integration and  $y_4$  indicated at 804 is the series with integration. The series  $y_3(t)$  at 802 was calculated with the following equation:

15

10

5

$$y_3(t) = \frac{y_2(t)}{\sigma[y_2(t)]}$$
 Eq. 4

where  $\sigma[y_2(t)]$  is the standard deviation in  $y_2(t)$  over a significant period before contact.

In contrast, the series  $y_4(t)$  is calculated by first computing a series,  $y_3(t)$  (not shown on FIG. 8) with the following equation:

$$y_3(t) = \int_{t-\tau}^{t} y_2(t)dt$$
. Eq. 5

25

The integration was performed numerically using point-to-point trapezoid rule. The increment or period,  $\tau$ ,

was set to 10 seconds. Using the  $y_3(t)$  series generated from Eq. 5, the series  $y_4(t)$  as indicated at 804 is generated with the following equation:

5 
$$y_4(t) = \frac{y_3(t)}{\sigma[y_3(t)]}$$
 Eq. 6

where  $\sigma[y_3(t)]$  is the standard deviation in  $y_3(t)$  from Eq. 5 over a significant time before contact. series and indicated 802 **У**3  $V_4$ at and respectively, are dimensionless. The values of y4 represent multiples of the standard deviation  $\sigma$  from y<sub>3</sub> or integral values. It is apparent from FIG. 8 that illustrated series  $y_4$  more clearly indicates engagement point than series y3. For this reason, integration is generally desirable due to cumulative effects.

10

15

20

25

Using the series  $y_4(t)$  indicated as 804, the "upper" limit or bound S2 of engagement was determined to be the first point for which  $y_4(t)$  exceeded 20. Thus, the threshold  $h_2$  described above was set at 20 (but could be set as low as 2 as desired). The "lower" limit or bound for the point of engagement  $S_1$  was determined by starting at S2 and working backward to the first point where the absolute value of  $y_4(t)$  is <1, i.e. threshold  $h_1=1$ . The point S was acquired from the first point after  $S_1$  where  $y_4$  exceeded 4, i.e. threshold  $h_s=4$ . All three points,  $S_1$ ,  $S_2$ , and S are illustrated on FIG 8 as 808, 806, and respectively. FIG. 9 generally illustrates the data

illustrated on FIG. 6, but graph 900 is an enlargement of data or measurements around the point of engagement S indicated as 602. It is noted that points  $S_1$  and  $S_2$  can be correlated across various series. For example,  $S_1$  and  $S_2$  correlate with 808, 908 and 806, 906 on FIGS. 8 and 9, respectively. Engagement point S correlates with 602, 810 on FIGS. 6, 8 and 9. Finally, it is noted that point  $S_1$  for which there is generally no discernable evidence of engagement or event onset can be obtained by any known means, including but not limited to the means described herein. The point  $S_1$ , however determined, is then used in the calculation of engagement point S and/or uncertainty.

10

Uncertainties corresponding to uncertainty 15 408 and block 542 module on FIGS. and respectively, were estimated for both force and displacement due to the uncertainty the determination of the point of contact. A rather conservative estimate of uncertainty can be obtained 20 by taking half the variation in both force displacement between  $S_1$  and  $S_2$ . Thus, uncertainty can be calculated as a function of  $S_1$  and  $S_2$ . This method yielded uncertainty estimates of 0.6µN and 5.6nm, Α respectively. more aggressive estimate 25 uncertainty is obtained by taking half the variation in force and displacement between the reading immediately before and after the point determined to be the point of engagement S. Thus, uncertainty can also be calculated as a function of S. This method

yielded uncertainty estimates in force and displacement of 0.077mN and 0.68nm, respectively.

The present inventions, including the method presented in FIG. 5, are advantageous over simple prior art threshold detection because significant event onset (e.g. engagement point) in experimental data can be detected by more sensitive means. The detection means or apparatus also includes a means for determining lower and upper bounds of the range of possible event onset, or  $S_1$  and  $S_2$ . The present inventions also include a means for determining the uncertainty in any test parameter due to the uncertainty in the determination of S. Finally, prior knowledge of the pre-event functionality of the data series of interest is not generally required.

10

15

20

Although the present invention has been described with reference to preferred embodiments, workers skilled in the art will recognize that changes may be made in form and detail without departing from the spirit and scope of the invention. For instance, although digitized signals are described herein for processing, analog signals and analog processing can be preferred as appreciated by those skilled in the art.